



Elemental quantification of large gunshot residues



A. Duarte^{a,b}, L.M. Silva^{a,b,c}, C.T. de Souza^a, E.M. Stori^a, L.A. Boufleur^{a,b}, L. Amaral^a, J.F. Dias^{a,b,*}

^a Ion Implantation Laboratory, Institute of Physics, Federal University of Rio Grande do Sul, Av. Bento Gonçalves 9500, P.O. Box 15051, CEP 91501-970 Porto Alegre, RS, Brazil

^b Graduate Program on Materials Science, Federal University of Rio Grande do Sul, Av. Bento Gonçalves 9500, CEP 91540-000 Porto Alegre, RS, Brazil

^c Forensic Institute of Porto Alegre, Av. Princesa Isabel 1056, CEP 90230-010 Porto Alegre, RS, Brazil

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ABSTRACT

In the present work we embarked on the evaluation of the Sb/Pb, Ba/Pb and Sb/Ba elemental ratios found in relatively large particles (of the order of 50–150 μm across) ejected in the forward direction when a gun is fired. These particles are commonly referred to as gunshot residues (GSR). The aim of this work is to compare the elemental ratios of the GSR with those found in the primer of pristine cartridges in order to check for possible correlations. To that end, the elemental concentration of gunshot residues and the respective ammunition were investigated through PIXE (Particle-Induced X-ray Emission) and micro-PIXE techniques. The ammunition consisted of a .38 SPL caliber (ogival lead type) charged in a Taurus revolver. Pristine cartridges were taken apart for the PIXE measurements. The shooting sessions were carried out in a restricted area at the Forensic Institute at Porto Alegre. Residues ejected at forward directions were collected on a microporous tape. The PIXE experiments were carried out employing 2.0 MeV proton beams with a beam spot size of 1 mm². For the micro-PIXE experiments, the samples were irradiated with 2.2 MeV proton beams of $2 \times 2 \mu\text{m}^2$. The results found for the ratios of Sb/Pb, Ba/Pb and Sb/Ba do not correlate with those stemming from the analysis of the primer.

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1. Introduction

Gunshot residues (GSR) constitute a relatively easy way to identify an event where a firearm was discharged. Usually, GSR are deposited in the vicinity where the firing event took place including surfaces, objects and the bodies of the shooter and the victim. Such particles are commonly of spherical shape with a diameter of few micrometers and are characterized by a mixture of elements present in the primer, projectile, gun powder, case and projectile. In particular, the primer plays an important role since it contains barium nitrate, antimony sulfide and lead styphnate [1], which are considered the fingerprints of such particles [2].

Despite several techniques like Neutron Activation Analysis (NAA) and Atomic Absorption Spectroscopy have been used to characterize GSR, the most popular one is the Scanning Electron Microscopy coupled with an Energy Dispersive Spectrometer (SEM–EDS) [3]. The systematic use of SEM–EDS systems in forensic sciences dates back to the seventies [4] and ever since it became the most powerful technique accepted in court for the analysis of GSR [5]. Around the same time, Particle-Induced X-ray Emission

(PIXE) begins to be explored as a potential technique for use in forensic sciences [6,7]. However, the use of broad beam to analyze GSR proved to be a drawback when compared to SEM results.

With the advent of the scanning microprobe capability together with better X-ray spectrometers brought new impetus to ion-based techniques applied to forensic sciences. Although μ -PIXE and SEM–EDS can be used as complementary techniques [8], features like sensitivity, bulk analysis capability and non-destructive character make PIXE and other ion beam techniques like μ -PIXE, Elastic Backscattering Spectrometry (EBS) and Nuclear Reaction Analysis (NRA) powerful tools for GSR analysis. In general, basic techniques like μ -PIXE and EBS have a potential use in the analysis of GSR [2,9], while several other ion-based techniques can be employed for a full assessment of the case under study [5].

Despite a huge volume of work dedicated to the study of GSR, little attention has been given to the study of relatively large particles ejected during the discharge of a firearm. Usually, the focus is laid on particles deposited in the hands or clothes of someone who discharged a firearm. In this case, relatively small particles are the most likely to remain in such places since large ones are easier to be removed by several processes. On the other hand, relatively large particles may be thrown on the floor or just stick to a nearby wall. Despite these particles cannot be used to incriminate someone, they could act as supporting evidence in a crime scene. In

* Corresponding author at: Institute of Physics, UFRGS, Brazil. Tel.: +55 51 3308 7248.

E-mail address: jfdias@if.ufrgs.br (J.F. Dias).

this context, the aim of the present study is to measure the elemental concentration of such particles ejected in the forward direction during the discharge of a firearm in order to check whether there is any correlation between these GSR and the primer of pristine cartridges.

2. Experimental procedure

In order to measure the primer, 30 CBC (*Companhia Brasileira de Cartuchos*) ogive lead (CHOG) type cartridges were carefully taken apart. The part concerning the primer was then irradiated with a 2.0 MeV proton beam for the PIXE measurements. The beam spot size was about 1 mm². Typical currents and irradiation times were 0.3 nA and 200 s respectively. The X-rays were detected by a Si(Li) detector placed at 135° with respect to the beam direction. The energy resolution of the detector is 150 eV at 5.9 keV.

For the shooting sessions, a caliber .38 Taurus revolver was employed using the same ammunition described above. The aim consisted of a cardboard screen placed 1 m away from the shooter. Several 0.03 m long microporous tape were placed around the aim in order to collect the residues ejected at forward directions. A shooting session consisted of three consecutive shots. After a shooting session, the tapes were collected and stored for further analysis. The gun was cleaned after each shooting session. In total, 5 shooting sessions were performed.

Micro-PIXE measurements were performed using the Oxford Microbeams[®] system operating in triplet mode. The proton beam current was about 100 pA while the beam energy was kept fixed at 2.2 MeV. Raster scans of 250 × 250 μm² were performed over medium size residues (from about 50 up to 150 μm across). X-rays induced in the samples by the proton beam were detected with a SDD detector placed a 135° with respect to the beam direction. The energy resolution of the X-ray detector was 135 eV at 5.9 keV. The irradiation time was 30 min for all 28 residues analyzed in this work.

Finally, the quantitative analysis of the X-ray spectra was carried out with the GUPIXWIN software [10]. The standardization procedure employed the NIST 2704 standard (Buffalo River sediment) and Micromatter[®] thin films standards. The analyses of all samples including GSR particles were carried out in the thick mode approximation since the beam was completely stopped within the samples. Despite the surfaces of the particles are not flat, the present work opted for a non pixel-by-pixel analysis since such information is not representative of the particles as a whole. The geometry of particles in the range of 100 μm may lead to wrong elemental concentrations particularly for soft X-rays. For that reason, focus was laid on the study of the L-shell transitions of Ba, Sb and Pb only. Moreover, the analysis of GSR assumed that these particles were homogeneous since the elemental maps showed a high degree of uniformity in terms of elemental distribution. However, some inhomogeneity could be observed. Therefore, the present results represent the average of the elemental concentration over the particles and are presented as relative ratios instead of absolute elemental concentrations.

3. Results and discussion

Different tapes were measured with PIXE in order to access their elemental content. One particular brand was chosen on the basis of the least contribution to the background at low X-ray energies. Fig. 1 depicts the X-ray spectra obtained from the primer and the microporous tape selected for the collection of the GSR. The tape spectrum represents an average over 5 independent measurements, while the primer spectrum is an average over 30 spectra. All spectra were normalized by their respective charges accumulated

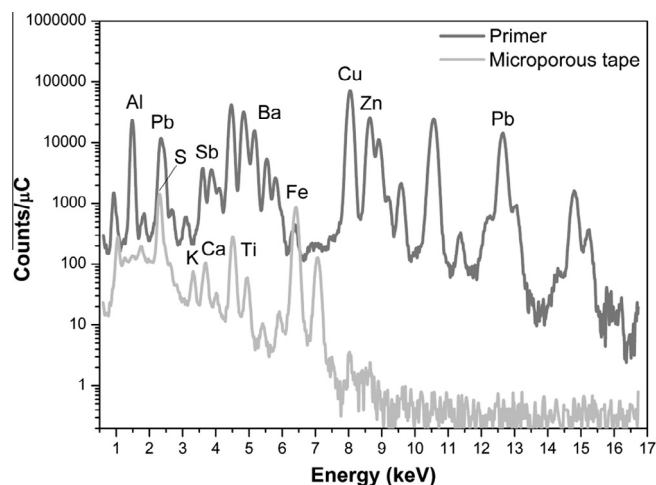


Fig. 1. X-ray yield of the primer (dark gray line) and the microporous tape (light gray line) as a function of the X-ray energy. The spectra were normalized by the charge accumulated during the experiments. The spectra shown for the primer and the microporous tape represent the average over 30 and 5 different analyses respectively.

Table 1

Elemental concentrations of the primer (30 samples) and the microporous tape (5 samples). All values are given in parts per million (ppm). The uncertainties represent the standard deviation of the mean based on the number of samples analyzed.

Element	Primer (n = 30)	Microporous tape (n = 5)
Na	–	$(5.4 \pm 0.3) \times 10^3$
Al	$(3.6 \pm 0.2) \times 10^5$	–
Si	–	$(5.2 \pm 1.2) \times 10^2$
S	–	$(5.4 \pm 0.3) \times 10^3$
K	–	$(2.5 \pm 0.3) \times 10^2$
Ca	–	$(2.1 \pm 0.3) \times 10^2$
Ti	–	$(3.2 \pm 0.2) \times 10^2$
Fe	$(3.1 \pm 0.2) \times 10^2$	$(4.1 \pm 0.5) \times 10^2$
Cu	$(2.5 \pm 1.0) \times 10^4$	–
Zn	$(1.0 \pm 0.4) \times 10^4$	–
Sb	$(6.7 \pm 0.6) \times 10^4$	–
Ba	$(2.9 \pm 0.1) \times 10^5$	–
Pb	$(2.4 \pm 0.1) \times 10^5$	–

Table 2

Elemental ratios of Sb, Ba and Pb found in the primer. These results were obtained from the analysis of 30 samples. SDM stands for standard deviation of the mean.

Ratio	Mean ± SDM (n = 30)
Sb/Ba	0.23 ± 0.02
Sb/Pb	0.28 ± 0.03
Ba/Pb	1.22 ± 0.08

during the experiments prior to averaging. The tape consists of a carbon matrix with considerable amounts of Na and S and other trace elements. The tape presents no traces of Sb, Ba and Pb. However, some attention must be paid to the amounts of S, Ca and Ti, since their K-shell transitions overlap with the L-shell transitions of Sb and Ba and with the Pb M-shell transitions. Although GUPIXWIN can deconvolute the overlapping peaks for all transitions, the results have larger uncertainties when overlapping peaks are analyzed. Therefore, the quantitative analysis were based on those transitions with the least contribution of the tape. In the particular case of lead, only L-shell transitions were considered for further analysis. The primer, as expected, is made of aluminum, antimony, barium and lead. The peaks concerning Cu and Zn stem from the cartridge case which lies around and beneath the primer. The

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